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#### 20 Abstract (Continued)

>100°C and peaked shortly thereafter, accompanied by significant description of water. At this point, rapid circuit detuning occurred and a very high reflected power level was maintained during the remainder of the heating process. Anhydrous molecular sieves containing CO2 do not absorb RF energy to any significant extent.

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# A Laboratory Study of the Dielectric Heating of Molecular Sieve Material BACKGROUND

In the current operational cycle of a carbon dioxide (CO<sub>2</sub>) removal system employing molecular sieve material, it is necessary at some point in the cycle to regenerate or desorb a large volume of molecular sieves by removing the CO<sub>2</sub> and water (H<sub>2</sub>O) molecules adsorbed in a previous step. The conventional regeneration procedure involves simple resistive heating of the sieve bed to a temperature in the range 200-370°C (400-700°F) and drawing off the carbon dioxide and water molecules that are released. In this procedure, the heat source (generally an electrical resistance element) causes the molecules of material to heat from the surface inward so that successive layers of molecules are heated. This is not advantageous in the case of molecular sieve material, since it is a poor thermal conductor and therefore requires a tight spacing (typically less than 2.5 cm) of the resistance elements. Even under optimum conditions, there is always the risk of damaging the surface material while attempting to heat the deeper layers.

An alternative approach is to employ the technique of radiofrequency (RF) or dielectric heating. Such a procedure is used in industry in the cases of poor electrical conductors which generally tend to be poor thermal conductors as well. In our case, molecular sieve material falls into both of these categories. This program was initiated to design and build a 1 KW dielectric heating device and to evaluate its performance in conjunction with a molecular sieve bed.

#### CONCLUSIONS AND RECOMMENDATIONS

A number of complexities are present in the dielectric heating/desorption of molecular sieve material. Several conclusions, however, can be made:

- 1. Water, because of its RF-energy absorbing capacity, is necessary in the dielectric heating of molecular sieve material.
- 2. As the moisture content of the sieve bed is decreased, the substrate becomes a more nonpolar, non-dissipative medium which results in circuit detuning and inefficient heating.

- 3. Assuming a constant  $\varepsilon \tan \delta$  value below  $100^{\circ}$ C, both the rate of heating (dT/dt) as well as the rate of  $CO_2$  desorption (dCO<sub>2</sub>/dt) of the molecular sieve bed are approximately linear functions of the input RF power (in kilowatts).
- 4. The highest temperature attainable under the present conditions (13.56 MHz) is not expected to be much above 100°C.
- 5. As many as three secondary sites of CO<sub>2</sub> absorption appear to be operative in the molecular sieve material.

Rapid sieve bed heating and CO<sub>2</sub> desorption occurred by using dielectric heating techniques but, utilization of such a device does not appear practical at this time. The requirement of water being present in the sieve material severely reduces the loading of CO<sub>2</sub> in the sieves and the eventual desorption of water results in inefficient operation of the RF generator.

Improvements in the operation of this technique would require initial drying of the air to enhance  ${\rm CO}_2$  loading before equipment optimization can be realized. Additional design and testing is also needed in the RF/dielectric engineering rather than in the chemistry aspect of the problem. The additional studies should include: a frequency analysis of both the dielectric constants and loss factors of the molecular sieve bed at various levels of  ${\rm H}_2{\rm O}$  and  ${\rm CO}_2$  content; the design of automatic frequency control (AFC) circuits for the on-line tuning of the matching network during the heating process; and the design of more effective  ${\rm Q}_{\rm L}$  circuits in order to minimize any energy loss in the tank coil. Only in this way can the impact of the various interrelated parameters be assessed to the fullest extent.

A second area which merits future consideration involves the choice of an alternative RF absorber to replace water. Ceramic materials are a potential solution but unfortunately, many of these have very low values of  $\varepsilon \tan \delta$  (< 0.01) at frequencies below 100 MHz. However, a special class of ceramic insulators, high K ferroelectrics, have abnormally high  $\varepsilon \tan \delta$  values and because of their crystal structure they have very high internal polarization values, thereby exhibiting very high effective dielectric

constants. The ferroelectrics are based on barium titanate (BaTiO $_3$ ) which has a peak dielectric constant of 6000 at the Curie point of  $120^{\circ}$ C. If future work in dielectric heating of molecular sieve beds is continued it should include testing BaTiO $_3$  dispersed in the molecular sieve bed as an alternative RF energy absorber.

#### RESULTS AND DISCUSSION

Dielectric heating is characterized by rapid, uniform heating and freedom from electrical and mechanical contact. The material to be heated is positioned between two electrode plates which are subjected to a rapidly alternating high voltage. In essence, the entire operation resembles that of a capacitor in which some energy loss occurs across the plates thereby causing a heating effect in the material spacing the plates. When subjected to a high frequency alternating voltage, the molecules in the material, acting like miniature dipoles, attempt to align themselves with the electrical field. Under the influence of this high frequency alternating electrical field, the particles oscillate about their axes, creating intermolecular friction which manifests itself as heat. All molecules are set in action at the same time. Thus, heat is generated quickly, uniformly — heat efficiencies are maximized. Power output from dielectric heaters ranges into the hundreds of kilowatts at frequencies of 1-150 MHz.

The electrical power, so transferred to the load as heat, is a function of the frequency, the square of the potential across the plates, and the dielectric properties of the material.

Heat Input (watts) =  $2 \pi f V^2 C \tan \delta$ 

where f = frequency, V = voltage, C - capacitance (= linear geometrical function of  $\varepsilon$  (dielectric constant)) and tan  $\delta$  = dielectric loss.

The dielectric strength,  $\varepsilon$ , and dielectric loss,  $\tan \delta$ , expressed as the product,  $\varepsilon \tan \delta$ , exert the greatest influence on the amount of heat generated for each material. Consequently, the larger the value of  $\varepsilon \tan \delta$  for a given material at a particular frequency, the more efficient will be the heating of that material. A few such values of  $\varepsilon \tan \delta$  are contained in Table I.

# Table I $\varepsilon$ tan $\delta$ at 10 MHz (20°C)

water (liquid, 25°C)	0.391		
anhydrous sieves (5A)	0.0348		
carbon dioxide (gas)	0.00		

It can be seen that water, by virtue of its highly polar nature, is a very effective energy absorber. Anhydrous molecular sieves, which are really crystalline metal aluminosilicates, are much less effective, while carbon dioxide, having no net dipole moment, will not absorb any electrical energy of any significance.

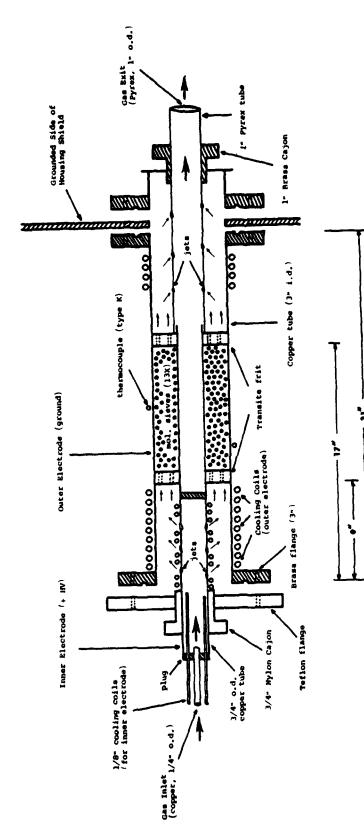
The point learned from such a comparison is that only in those cases where a molecular sieve bed loaded with carbon dioxide contains a significant amount of water will the dielectric heating process be energy-efficient.

#### Device Design and Operation

#### Capacitor

A schematic of the capacitor for containment of the molecular sieve material is presented in the following figure. Basically, a coaxial cylindrical arrangement was chosen; the inner/outer electrode diameters being 3/4 in. and 3 in., respectively. The effective length of the capacitor could be varied by simply moving the transite frit spacers closer or farther apart. In the experiments described here, however, a standard length of 8 in. was chosen. This permitted approximately 0.5 Kg of molecular sieves (weighed at room temperature, 60% RH) to be used as the interelectrode dielectric medium.

The cylindrical electrodes were electrically isolated from each other by a Teflon flange which incorporated a 3/4 in. Nylon cajon fitting in order to effect a high voltage vacuum seal with the inner copper electrode. The inner electrode was also water-cooled at that end closest to the Teflon flange in order to prevent excessive heat conduction to the Nylon cajon.



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Capacitor Schematic

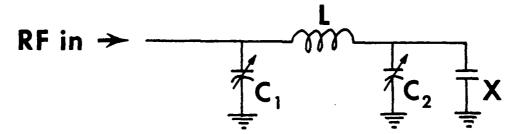
Similarly, the ends of the outer copper electrode were water-cooled via 1/4 in. copper coils in an effort to minimize excessive heating of the "O"-ring flange seals.

The inner electrode was terminated after the last transite frit (right side of figure) and electrically insulated from the terminal brass flange by a concentric coaxial length of 1 in. o.d. Pyrex tubing. Hot gases exiting from the molecular sieve bed were admitted to the Pyrex tube by an array of radial ports or jets and pumped away to a conventional vacuum line for analysis.

Two separate lengths of 1/8 in. copper tubing were used as thermocouple shields and each terminally sealed and soldered at one end to the atmospheric side of the outer copper electrode at points midway and one-quarter of the distance between the two transite frits. The remaining portions of the separate tubing sections were grounded and brought out through the housing shield by standard 1/8 in. Swagelock bulkhead fittings. Separate, fast-responding thermocouples (type K) were used to monitor the temperature in both regions.

#### Circuit

After a number of trial studies to determine the most reliable circuit for coupling the RF energy to the capacitor, a  $\pi$ -LC arrangement was chosen as the matching network.



The loading and turning capacitors,  $C_1$  and  $C_2$ , respectively, were vacuum dielectrics rated at 10 KV/42 A and continuously variable over a range of 10-500 pf (Dolinko and Wilkins, Inc., Union City, N.J.). The tank coil, L,

was comprised of 1-1/2-2 turns of 3/4 in. diameter copper tubing on a 7 in. form with 1/2 in. spacings between turns. Like the coaxial cylindrical capacitor (X) containing the molecular sieves, the tank coil was also water-cooled. Electrical contact between the various components was effected by 3/4 in. wide strips of 20-gauge sheet copper which had been silver-plated in order to maximize the effective  $Q_L$ . The entire circuit was contained in a grounded housing (3'x3'x4') which was fabricated from 1/8 in. thick sheet aluminum.

The inner copper electrode of the molecular sieve capacitor was connected to the high RF potential while the outer electrode was grounded to the housing shield as were both thermocouple shields. A whisperjet ventilation fan was situated in the base of the shielded enclosure below the molecular sieve capacitor. Its primary function was to effect rapid cooldown of the molecular sieve bed between experiments.

#### RF Generator

A 10-kilowatt RF Generator (Model HFS-10,000D, Plasma-Therm, Inc., Kresson, N.J.) was employed as the source of RF energy which was subsequently coupled to the circuit containing the molecular sieve capacitor by a standard heavy-duty 50 ohm coaxial cable. An industrial/medical frequency of 13.56 MHz was employed in all experiments at input power levels of 0.5-1.5 KW continuous. The incident and reflected power levels were continuously monitored by an in-line SWR meter (SWR = standing wave ratio). In the majority of cases described here, an automatic power control pilot circuit was used to maintain a relatively constant input power level. In addition, an automatic shutdown capability was always used in order to prevent damage to the power tube (high  $\mu$ -triode) at excessively high SWR levels.

#### Gas Flow

The incoming gas-mixture to the molecular sieve bed was regulated in a stepwise process by:

- (a) flowing an air-0.2% CO $_2$  standard gas mixture at a rate of  $\sim 7$  SCFH through a thermostatted distilled water reservoir held at a temperature slightly below ambient, thereby saturating the gas stream (i.e., 100% RH). RH = relative humidity:
- (b) passing the saturated gas stream through a series of heat exchange coils maintained at a constant ambient temperature;
- (c) maintaining the temperature difference ( $\Delta T$ ) of the two reservoirs to that particular psychometric value necessary to produce a constant relative humidity of 85%:
- (d) monitoring the final RH of the gas in order to make any necessary temperature corrections.

By such a procedure, the relative humidity of the gas  $(0.2\% \text{ CO}_2\text{-air})$  entering the molecular sieve capacitor was monitored and maintained at a constant 85% RH. After exiting from the molecular sieve bed, the gas flow was sampled continuously by an in-line infrared  $\text{CO}_2$  gas analyzer (Beckman Model No. 865) in an effort to determine the degree of sieve loading.

#### Desorption of Molecular Sieve Bed

After appropriate sieve loading, dry nitrogen gas (at a flow of 5 SCFH) was admitted to the sieve bed which was then heated dielectrically. The exit gas (enriched in desorbed  ${\rm CO_2}$  and  ${\rm H_2O}$ ) proceeded through a dry ice trap (to remove water), the infrared  ${\rm CO_2}$  gas analyzer and a final trap cooled to liquid nitrogen temperature for collection and subsequent manometric measurement of total  ${\rm CO_2}$  content. Both the sieve bed temperature and the exit gas  ${\rm CO_2}$  levels were monitored continuously during the entire heating/desorption process and registered on conventional millivolt chart recorders.

After conducting a number of trial-and-error experiments in order to ascertain appropriate flow rates, loading conditions, and RF input power levels, a total of eleven sequential loading/heating/desorption cycles were initiated. The first ten experiments (Nos. 1-10) were conducted with 0.2%

 ${\rm CO}_2$ -air gas mixtures at 85% RH (Relative Humidity). Run No. 11 was conducted with a 0.2%  ${\rm CO}_2$ -air gas mixture at 0% RH. In every run a quantity of 0.2%  ${\rm CO}_2$ -air tank gas mixture in excess of that required for complete  ${\rm CO}_2$  saturation ( ${\sim}3\%$ ) was employed. The quantity of  ${\rm CO}_2$  actually loaded on the sieve bed, however, was determined from integration of the exit gas differential  ${\rm CO}_2$  (%) vs. time curves to the breakthrough point.

After appropriate sieve loading, the RF field was applied to the capacitor which was continuously swept with a stream of dry nitrogen at a rate of 5-7 SCFH. This was found to be necessary in order to retard plasma formation which occurred at the lower pressures (< 300 torr). The  $\rm CO_2$  level (%) in the gas exiting the capacitor was monitored continuously during the heating cycle as was the temperature of the outer electrode. This is illustrated in the following figures (Nos. 1-11) for the eleven experiments reported here. The total quantity of  $\rm CO_2$  desorbed was determined by two methods: integration of the exit gas  $\rm CO_2$  (%) level vs. time curves and static collection and manometric measurement of the total  $\rm CO_2$  quantity desorbed. The data are summarized in Table II.

In Table III are summarized the average values for the time rate of temperature rise (dT/dt),  $\rm CO_2$  level in the exit gas (dCO<sub>2</sub>/dt) and appearance times of the largest  $\rm CO_2$  peak as a function of input RF power for three power levels. Input power settings higher than  $\sim 1.5$  KW proved to be intractable for the amount of sieve material (447 g) used in these experiments.

#### Tuning

In order to efficiently couple the energy of the RF field (13.56 MHz) to the capacitor containing the molecular sieves (447g-13X), the  $\pi$ -LC matching network had to be properly tuned to minimize the standing wave ratio (SWR) or reflected power. This was accomplished by adjustment of the loading and tuning capacitors,  $C_1$  and  $C_2$ , respectively. After several trial-and-error experiments, it was observed that  $C_1$  could be set to a relatively constant value whereas  $C_2$  had to be adjusted slightly during the course of the RF heating cycle in order to maintain a minimum reflected power level.

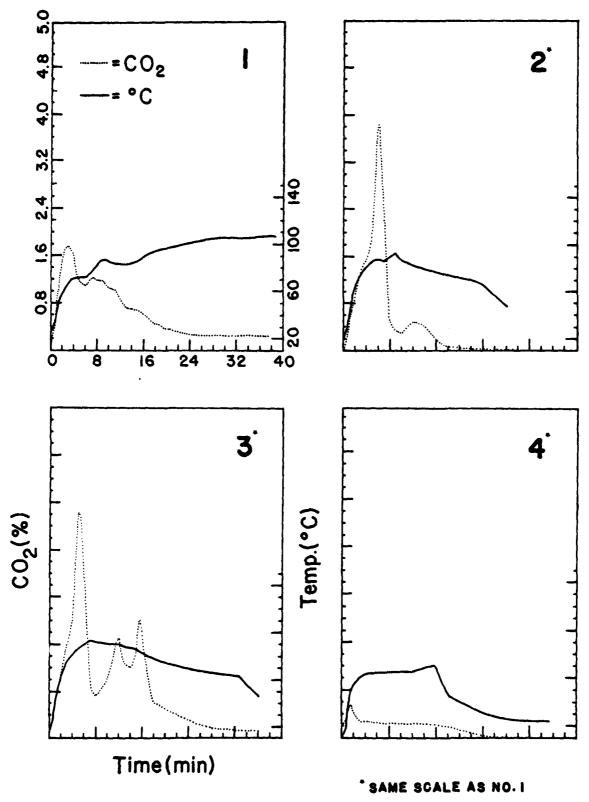


Fig. 1-4 -  $CO_2$  levels in exit gas

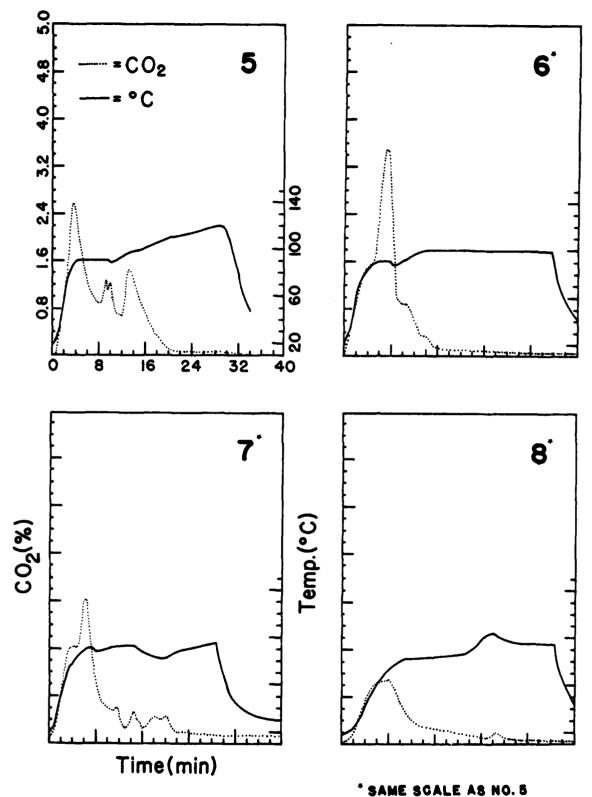


Fig. 5-8 - CO<sub>2</sub> levels in exit gas

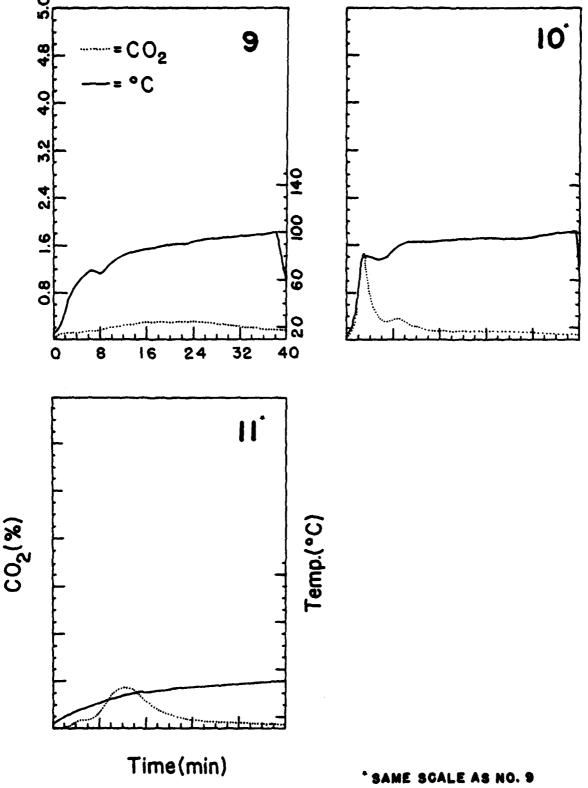


Fig. 9-11 - CO<sub>2</sub> levels in exit gas

Table II. Temperature and CO<sub>2</sub> description rates of Type 13% Molecular Sieves (447 g, M. R. Grace Co.) Loaded with 0.2% CO<sub>2</sub>-air (85% RH) and heated dielectrically at 13.56 MHz (0,5-1.5 KM).

Appearance times CO;-maxima (min)	2.95	6.32	5.16	1.29	3.61	7.19	6.29	6.95	20.80	2.83	12.85	
dc02/dt (% c02/min)												
CO <sub>2</sub> Desorb/Coll	0.653	0.710	0.843	0.746	0.724	0.597	0.757	0.675	0.778	0.643	0.716	
Collected	1.104	1.022	1.390	0.225	1.186	1.349	0.838	0,634	0.429	0.552	0.532	
CO <sub>2</sub> c Described (9)	0.721	0.726	1.173	0.168	0.859	908.0	0.635	0.428	0.334	0.335	0.381	
dT/dt max (°C/min)	24.8	31.8	33.7	34.8	36.8	34.0	31.1	10.7	18.8	45.1	2.0	
Max (C)												
RP Power (KW)	~	~	7	-	-	-	~	0.5	1.50	1.5	1.0	
CO2 CO Loaded (9)	0.694	0.826	ъ	1.479	0.687	1.0%	3.206	0.522	0.646	0.332	0.690	
Total <sup>b</sup>	15.76	17.68	17.09	16.74	18.49	19.22	16.31	12.71	13.43	10.73	23.69	
Tank No.	~	~	m	•	s	9	,	1+6	3+4	2+5	<b>3</b> 0	
sp. <u>Load</u>	CO2 + H2O	•		•	:	•	•	•	•	1	ී ග	
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0.2% CO2 in air at Of Relative Humidity

Dyotal quantity of CO2 in O.2% air mixture passed over sieves

CDetermined from integration of breakthrough curves

dyalue omitted due to recorder malfunction

<sup>e</sup>Untuned

feasured manometrically

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Table III. Average Values of Rates of Heating and CO<sub>2</sub> Desorption of Type 13X Molecular Sieves Heated Dielectrically at 13.56 MHz.

RF Power (KW)	dT/dtmax (°C/min)	dCO <sub>2</sub> /dt (% CO <sub>2</sub> /min)	Appearance times CO <sub>2</sub> -maxima (min)		
0.5	10.7	0.316	6.95		
1.0	32.4	0.699	4.68		
1.5	45.1	0.924	2.83		

During the initial minutes of a heating cycle, very low reflected power readings were observed. For example, at the 1 KW power level, the initial I/R readings (I = incident, R = reflected power) were of the order of 1.0/0.05. A rapid temperature rise ensued and the CO, level in the exit gas began to rise rapidly. After several minutes, the temperature of the molecular sieve capacitor plateaued out near 100°C and the CO, concentration in the exit gas reached its maximum value. At this time, there was also a significant amount (~15 g) of water desorbed from the capacitor and subsequently collected in the dry ice trap. Very shortly thereafter (< 2 min), the entire circuit detuned and could not be retuned to the initial low reflected power level. For example, in the 1 KW runs, the optimum I/R values after the maximum CO, wave were of the order of 1.0/0.6. This effect was observed in every experiment where the initial reflected power level was low. Presumably, the loss in absorbed water upon heating renders the molecular sieve bed a more nearly perfect and hence non-dissipative capacitor. In the extreme limit, anhydrous molecular sieves have a Etanô value observed for a material like glass-bonded Mica ( $\infty$ .01), a widely used low loss, high voltage, capacitor medium.

#### Temperature

The data in Table III readily demonstrates that the temperature of the capacitor, which closely approximates the temperature of the molecular sieve bed rises at a faster rate as the input RF power level is increased. The temperature eventually approaches a plateau level near  $100^{\circ}\text{C}$  ( $T_{\text{max}}$ ) and is maintained nearly constant for the remainder of the heating cycle. The capacitor must be properly coupled to the input RF field so as to absorb energy and hence experience a temperature rise. This is illustrated in the case of Run No. 9 where, because of an impedance mismatch, a rather low dT/dt resulted even though a high input power level was employed. A similar result was also observed in Run No. 11, although in this case, the impedance mismatch is believed to be a direct result of the absence of significant amounts of initial moisture in the sieve bed.

### CO<sub>2</sub> Desorption

During the RF heating cycle, the CO, level (%) in the exit N, purge gas rose dramatically as the temperature of the molecular sieve bed approached its plateau level near 100°C. After reaching some maximum value, the CO, level then diminished rapidly and was shortly accompanied by a significant quantity of desorbed water. Simultaneously, or as near so as could be determined, the entire circuit detuned as evidenced by a high reflected power reading. The temperature of the sieve bed, however, was still maintained at or near 100°C. In those cases where the sieve temperature plateaued at much below 100°C (Exp. Nos. 4 and 11), a much smaller initial  $^{\mathrm{CO}}_{2}$  peak was observed. Apparently, temperatures at or above  $100^{\mathrm{o}}\mathrm{C}$  are necessary for rapid  ${\rm CO}_2$  desorption. In several experiments it was observed that several secondary CO, maxima (1-3) appeared at much later times (8-20 min.) in the heating cycle after detuning had occurred. These could only be attributed to secondary CO, absorption sites in the molecular sieve bed which are apparently distinct from and minor to the primary site of CO, absorption. The rate of initial  ${\rm CO_2}$  desorption,  ${\rm dCO_2/dt}$ , increased with increasing RF power as expected, while the appearance times of the first CO, peak were shortened. Moreover, as can be seen in Table III, both the desorption rate,  $dCO_2/dt$ , as well as the temperature rise, dT/dt, appeared to increase in an approximately linear fashion with the RF input power (kilowatts).

#### Effects of Moisture

As mentioned earlier, it is essential that moisture be present in the sieve bed in order for the capacitor to absorb energy and thus heat up and desorb any  ${\rm CO}_2$  present. This was borne out by the results of Exp. No. 11 where moisture was intentionally excluded from the 0.2%  ${\rm CO}_2$ -air gas stream used to initially load the sieve bed with  ${\rm CO}_2$ . As expected, both the maximum temperature attained ( ${\rm T}_{\rm max}$ ) and the rate of temperature increase (dT/dt), as well as the rate of  ${\rm CO}_2$  desorption (dCO $_2$ /dt), were noticeably diminished.

Water has both a high dielectric constant ( $\epsilon$ ) and high loss factor (tanô) at 13.56 MHz which is a direct result of the highly polar nature of

its 0-H bonds. Water, as a miniature dipole, tends to react to the applied RF field and align itself accordingly. As the applied field changes at a very rapid rate (13.56 MHz), all the elastic distortions, deformations, and displacements which occur under stimulation from the field and the restoration forces involved manifest themselves as heat. Energy absorption from the RF field is sharply curtailed (in the absence of this very dipolar medium), thereby resulting in high reflected power levels and greatly reduced rates of heating and desorption. Indeed, this is precisely the reason for the detuning of the molecular sieve capacitor after the temperature plateau of  $\sim 100^{\circ}$ C is reached. Shortly after reaching  $100^{\circ}$ C or theresbouts, a significant quantity of water is desorbed from the sieve bed, thereby rendering the capacitor less dipolar in nature and therefore less able to absorb energy from the RF field.

A second effect of the presence of water in the sieve bed concerns its ability to be preferentially absorbed on the molecular sieve substrate; much more so than carbon dioxide which has a direct bearing on two aspects of the experiments conducted here. First, in those cases involving sieve loading at the recommended high humidity level (85% RH), a rather small percentage of  $\rm CO_2$  was absorbed from the loading gas stream. Secondly, during the dielectric heating process, the  $\rm CO_2$  level (%) in the exit gas rose dramstically as the temperature approached  $\rm 100^{\circ}C$ . Both effects can be explained by the selective sieve absorption of  $\rm H_2O$  and concomitant displacement of  $\rm CO_2$ . The second effect is believed to occur by sieve desorption of  $\rm H_2O$  (which occurs near  $\rm 100^{\circ}C$ ) and dynamic reabsorption/displacement at those sieve sites containing absorbed  $\rm CO_2$ , thereby resulting in a high exit gas  $\rm CO_2$  level as the temperature approaches  $\rm 100^{\circ}C$ .

